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The Evaluation of Nanoemulsion Cinnamon Extract (*Cinnamomum burmanii)* on Lipcream Preparation as a Natural Dyes

Eriska Agustin* and Maulia Agustina
Faculty of Pharmacy, Universitas Kader Bangsa, Palembang, Indonesia
*Corresponding author: Eriska Agustin | Email: eriska.agustin@gmail.com

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Abstract : The use of nanoemulsion in the cosmetic field has been widely developed, one of which is the use of natural dyes from natural ingredients. Nanoemulsion has the ability to provide a wider variety of colors due to its high solubilization and translucent nature. The effort to produce natural dyes can be obtained form natural resources, one of which is the cinnamon plant. Beside having a distinctive aroma, cinnamon can also produce a brownish pigment that comes form the cinnamaldehyde compound. In this study, Cinnamon extract samples were obtained by maceration method using ethanol solvent pa, cinnamon extract nanoemulsion preparations were made using a sonicator bath for 60 minutes, and lipcream preparations were made using the trituration method. The results showed that the cinnamon extract from formula F1 nanoemulsion had a particle size of 273±0.032 nm. Evaluation of the lipcream preparations carried out included organoleptics which produced a distinctive aroma of cinnamon, the resulting color was softer brown compared to conventional lipcream preparations, the texture was soft and homogeneous, the resulting pH test met the pH requirements of the lip cream, namely 4.93-5.02, adhesion 5-6 cm and 4-5 seconds of adhesion ($p>0.05$). The cinnamon extract nanoemulsion preparations produced particle sizes that entered the nano range and lipcream preparations produced color pigments that were softer than conventional preparations.

Keywords: Cinnamaldehyde, Cosmetic, Cinnamon, Dye, Nanoemulsion

1. INTRODUCTION

Indonesia as a country that has a lot of natural wealth, especially plants that have many benefits, one of which can be used as a natural dye. The use of natural dyes minimizes the use of synthetic materials which have a higher risk [1]. Efforts to produce natural dyes that are environmentally friendly can be done by extracting plant parts that contain natural pigments. One of the plants that has natural pigments is cinnamon [2]. Cinnamaldehyde is the main compound in cinnamon bark and is polar [3]. The more cinnamaldehyde extracted, the darker the resulting color. Cinnamon extract produces a light brown to dark brown color [1]. In the previous research, cinnamon extract was used to make nanoemulsion. The results showed that the smallest globule size of cinnamon extract nanoemulsion was 24.2 nm [9]. Natural dyes produced by plants have several obstacles, including impractical processes, limited color variations, low homogeneity, the tendency for extracts of natural ingredients to be unstable in the long term, so they cannot last long [5]. The development of nanoemulsion delivery systems for cosmetics aims to increase the stability of dye stuffs, high

homogeneity, the use of small concentrations can provide good color, the right consistency for skin application, and comfort on the skin. Apart from that skin irritation or side effects are very minimal [4]. This natural dye can be used as a cosmetic to enhance your appearance to make it more attractive, one of which is by using lipcream preparations to color your lips. Lipcream can moisturize the lips longer than the solid form because it has a fairly high oil content and provides a more even lip color [7].

In this research, cinnamon extract nanoemulsion was prepared with the aim of evaluating the size of the globules produced by cinnamon extract nanoemulsion. Nanoemulsion carriers are solid spherical particles with an amorphous, lipophilic surface. Although the system is thermodynamically unstable, it can be stabilized through the use of emulsifying agents. Typically, nanoemulsion are composed of oil, an emulsifier, and water-based phase [6].

This study also wanted to evaluate lipcream formulas made with viscous extracts and extracts made from nanoemulsions. Preparation of nanoemulsion using the Top Down method with the addition of an emulsifier with 15% extract using a sonicator bath to produce a translucent natural dye which will later be incorporated into the lipcream preparation so that it can be applied to the skin. The main compound of cinnamon extract is polar, so the nanoemulsion formula in cinnamon extract is expected to increase the stability of lipcream preparations whose composition is oil in water. Evaluation of the physical quality of nanoemulsion preparations will be carried out by organoleptic and particle size tests, while lipcream preparations will evaluate organoleptic, pH tests, adhesion, spreadability, and preparation stability.

2. MATERIALS AND METHODS

2.1. Materials

In this study, the tools and materials used included cinnamon powder obtained from the city of Bandung, ethanol pa (Merck), white wax (Brataco), bees wax (Brataco), cetyl alcohol (Brataco), kaolin (Brataco), tocopherol (Brataco), methyl paraben (Brataco), Propyl Paraben (Brataco), titanium dioxide (Brataco), dimethicone (Brataco), castor oil (Brataco), tween 80 (Brataco), Virgin Coconut Oil (VCO), Poliethylene glycol (PEG) 400 (Brataco), aquadest (Brataco),. The tools used for this research were rotary evaporator (Eyela Digital Water Bath SB-1000), Particle Size Analyzer (PSA) (Horiba SZ 100), magnetic stirrer, sonicator bath, and other laboratory equipment.

2.2. Cinnamon Extract Preparations

In the process of making Cinnamon extract preparations, a modified procedure was carried out from research conducted by Azkiya [8]. The dried cinnamon simplicia is sieved using 100 mesh size. As much as 600 g of chopped cinnamon powder was carried out by the extraction process using the maceration method with ethanol pa 1:6. Extraction was carried out in 3 cycles, one cycle for 3 days. Every day occasional stirring is carried out and each cycle is re-maceration carried out with a new solvent. The results of the liquid extract were concentrated using a rotary evaporator to obtain a thick extract.

2.3. Cinnamon Extract Nanoemulsion Preparations

In this study, a cinnamon extract nanoemulsion formula was prepared based on a modification of Purwandari's research [9] by optimizing the formulation with the following ingredients:

Table 1. The Cinnamon Extract Nanoemulsion Formula Design

Ingredients	Concentration (%)			
	F0	**F1**	**F2**	**F3**
The Cinnamon Extract	0	15	20	25
Tween 80	20	20	20	20
Virgin Coconut Oil (VCO)	4	4	4	4
Polyethylene Glycol (PEG 400)	10	10	10	10
Aquadest	ad 100	ad 100	ad 100	ad 100

The nanoemulsion preparation process begins with weighing each ingredient and mixing each phase. The VCO, Tween 80, and cinnamon extract were first put into a beaker, then stirred until homogeneous using a magnetic stirrer for 15 minutes. Next, PEG 400 was added to the beaker and stirred again for 10 minutes. All of these mixtures were reduced in particle size using a sonicator bath for 60 minutes. After the nanoemulsion is formed, the aquadest material is added and stirred using a magnetic stirrer for about 15 minutes [10]. After that, each formulation was evaluated for the particle size and physical stability to determine the best formula.

2.4. Cinnamon Extract Nanoemulsion Preparations in Lipcream

In this study, lip cream preparation was made using 2 ingredients, namely conventional cinnamon extract and nanoemulsion extract. The following ingredients are used to make lipcream preparations :

Table 2. The Cinnamon Extract Nanoemulsion Formula Design

No	Ingredients	Concentration (%)		
		F0	F1	F2
1	Cinnamon extract	-	15	-
2	Nanoemulsion extract	-	-	15
3	White wax	6	6	6
4	Bees wax	3	3	3
5	Cetyl Alcohol	2	2	2
6	Dimethicone	5	5	5
7	Kaolin	3	3	3
8	Tocopherol	0.05	0.05	0.05
9	Methyl Paraben	0.18	0.18	0.18
10	Propyl Paraben	0.02	0.02	0.02
11	Titanium dioxide	2	2	2
12	Castor Oil	ad 100	ad 100	ad 100

The process of making lipcream begins with weighing all the ingredients. Then, the oil phase is melted using a steam cup at a temperature of approximately 70°C. Oil phase ingredients include white wax, beeswax, cetyl alcohol, castor oil, and dimethicone. The melted mixture is transferred to a mortar and then crushed and added to the aqueous phase, including tocopherol, titanium dioxide, methyl paraben, propyl paraben, until a creamy mass is formed. Furthermore, the cinnamon nanoemulsion extract is added to the lipcream base while stirring until homogeneous [11]. The conventional lipcream preparations also use the same ingredients, but the active ingredient used is cinnamon extract which is not made of nanoemulsion.

2.5. Evaluation of Cinnamon Extract Nanoemulsion

In this study, evaluation of the physical quality of cinnamon extract nanoemulsions was carried out, including the size of the nanoemulsion globules using the Particle Size Analyzer (PSA). The sample is put into the cuvette up to the specified mark and it is ensured that there are no bubbles. Samples were read 3 times to ensure consistent values.

2.6. Evaluation of Lipcream Cinnamon Extact Nanoemulsion

Evaluation of Lipcream preparations was carried out on two preparations, namely lipcream preparations using cinnamon extract nanoemulsions and conventional lipcream preparations. The following evaluations were carried out:

2.6.1. Organoleptic Test

The organoleptic observation involves an objective assessment of the texture, color, and scent of lip cream product. This test is conducted by examining these characteristics through direct observation [12].

2.6.2. pH Test

The pH test aims to determine the level of cidity or alkalinity in a formulation. For human lip skin, the normal physiological pH ranges between 4.0 and 6.0. The measurement is performed using a pH meter, which is first calibrated with buffer solutions of pH 7.0 (neutral), pH 4.0 (acidic), and pH 9.0 (alkaline). The concentration 1% of the test sample is prepared, and the elcetrode is immersed in the solution. The reading is taken once the pH valuse stabilizes. Each formula is tested in triplicate [12].

2.6.3. Homogeneity Test

The homogeneity test is conducted to assess whether the component in a formulation are evenly mixed. This test involves applying the product onto a transparent glass surface, then covering it with another glass plate. The preparation is considered homogeneous if no coarse particle or uneven areas are visible on the glass surface [12].

2.6.4. Spreadability Test

The spreadability testing is conducted to assess how easily and quckly the lip cream spreads during application, which affect user convenience. In this test, 0,5 g of the lip cream sample is placed on the glass surface lined with graph paper, and petri dish is placed over it for 60 seconds. The spread area is measured. Next, the formulation is sandwitched between two glasss plates, 200 g weight is applied for 60 seconds, and diameter of the spread is recorded [8].

2.6.5. Stickiness Test

The adhesion test is used to determine how long the lip cream adheres to the lips. The higher adhesion value indicates the longer sticking time. In this test, 0,5 g of the formulation is applied to a glass slide with a known surface area, then covered with another slide and pressed with 50 g weight for 50 minutes. The slides are then mounted on the testing device, and 20 g load is applied. The time taken for the two slides to separate is recorded as the adhesion value [8].

2.6.6. Physical Stability Test

The physical stability test is carried out to see if the sample preparation still has the same properties and characteristics for a certain period of time. The stability test was carried out by storing the sample preparations at room temperature and then observing organoleptic, pH, stickiness, spreadability, and homogeneity on days 0, 7, 14, and 28 [13].

2.6.7. Data Analysis

The research data were analyzed using both descriptive and statistical methods. Statistical analysis was performed using the one-way ANOVA technique. The p-value greater than 0.05 indicates that there is no significant difference in the effect of the variables among the formulas. Conversely, the p-value less than 0.05 suggests the significant difference, which is attributed to the variation in the concentration of Tween 80 sufactant used in the study.

3. RESULTS AND DISCUSSION

In this study, the yield of cinnamon extract was 17% from the maceration process using ethanol pa. The compound expected from cinnamon as a natural dye is cinnamaldehyde [9]. Cinnamaldehyde is the main compound in cinnamon bark and is polar in nature, consisting of a phenyl ring attached to an aldehyde group. the aldehyde carbonyl group is relatively strong polar [3].

The manufacture of nanoemulsion preparations, the composition of the materials used include VCO as the oil phase, Tween 80 as an emulsifier, and PEG 400 as a co-solvent. From the results of the globule size test using PSA, the globule size was 273 ± 0.032 nm in formulation F1 and 73 ± 0.069 nm for F0 (base). In the formulation F2 and F3, particle size testing was not carried out beacuse on the first day after making the nanoemulsion, the formulation experienced separation and thickening, so it was assumed that F2 and F3 were unstable. The nanoemulsion preparation from F1 looks transparent and the color is not too intense compared to the thick extract. The diameter of the globule size in the nanoemulsion formulation is still in the nano range. This is due to the composition of the use of surfactants and co-surfactants in the preparation. The type and percentage of emulsifier can affect globule size to stabilize the formation of droplets [14]. Therefore, the F1 formulation was chosen as the nanoemulsion formula incorporated into the lip cream preparation.

The size of the nanoemulsion globule enters the nano range, which is between 200-500 nm [15]. This is due to the effect of using a high emulsifier concentration so that the globule size becomes small and stable. The polar nature of cinnamaldehyde is also aided by an emulsifier that has a high HLB value, which makes the nanoemulsion preparation stable. A balanced mixture of the oil, water, and emulsifier phases can produce nanoemulsions with a clear and stable appearance, so it is hoped that this nanoemulsion preparation can be incorporated into lipcream preparations [6].

Figure 1. The Cinnamon extract nanoemulsion preparations F1 (left), Nanemulsion base (right)

The process of making conventional lipcream preparations and cinnamon extract nanoemulsions uses the same ingredients, the only difference being the composition of the extract. The concentration of each extract is 15% and the manufacturing process uses the trituration method.

The results of organoleptic observations show that conventional lip cream preparations have a semi-solid texture, dark brown color, and a distinctive aroma of cinnamon. Meanwhile, nanoemulsion lipcream preparations produce a light brown color and are softer than conventional lipcream preparations with the same amount of concentration, semi-solid texture, and a distinctive aroma of cinnamon. Based on statistical tests from the results of testing for 28 days stored at room temperature, there were no significant differences and changes in preparation for each preparation ($p > 0.05$) [16].

Figure 2. Lipcream Base Preparation

Figure 3. Lipcream Extract conventional (left), Nanoemulsion lipcream (right)

The evaluation of the pH of conventional lipcream preparations and nanoemulsions was carried out using a pH meter. The results of each test were carried out 3 times to ensure a constant value. The pH range obtained from the test results is between 4.93-5.02 respectively. Based on these results, the pH value still entered the allowed lip skin pH range and there was no significant difference from the pH value between conventional lipcream preparations and nanoemulsion lipcream tested on days 0, 7, 14, and 28 at room temperature storage ($p > 0.05$).

Table 3. The result of Testing the pH of Lipcream Preparations

Formula	0 Day	7th Days	14th Days	28th Days
F0	5.0 ± 0.005	4.95 ± 0.005	5.0 ± 0.006	4.94 ± 0.006
F1	4.95 ± 0.004	4.95 ± 0.006	5.0 ± 0.006	5.01 ± 0.006
F2	5.02 ± 0.001	5.0 ± 0.005	5.02 ± 0.006	5.01 ± 0.006

This shows that the lipcream preparations made are safe and do not cause irritation to the lips. the more alkaline or the more acidic the material is exposed to the skin of the lips, the more difficult it will be for the skin of the lips to neutralize and the skin can become dry, cracked, sensitive, and

susceptible to infection [12]. Therefore, the pH of cosmetics should be the same or as close as possible to the physiological pH of the skin [7].

The homogeneity testing is carried out to see whether the mixing of each ingredient is uniform and good or not. In conventional lipcream preparations, the results obtained are that the preparation appears brown in color and brown spots are visible in the preparation. This could be due to the fact that the viscous extract obtained was not homogeneous and only went through a coagulation process so that the fine simplicia powder was likely to be filtered out. Whereas nanoemulsion lipcream preparations produce homogeneous preparations and there are no fine spots even though the resulting color is lighter than conventional lipcream preparations [17]. This could be caused by the color resulting from making the nanoemulsion clearer and more transparent than the thick extract due to the influence of the addition of emulsifiers and other supporting materials which make the cinnamon extract nanoemulsion preparations perfectly dispersed [15].

Table 4. The result of Testing the Spreadability of Lipcream Preparations

Formula	0 Day	7th Days	14th Days	28th Days
F0	5 ± 0.005	5.0 ± 0.000	5 ± 0.001	5 ± 0.002
F1	5 ± 0.001	6.0 ± 0.005	6 ± 0.001	6 ± 0.006
F2	5 ± 0.001	5.0 ± 0.005	5 ± 0.000	6 ± 0.000

The spreadability and adhesion tests were carried out to evaluate the spread of the lipcream and the duration of its adherence to the lips. The results obtained were the spread of lipcream on average 5-6 cm and there was no significant difference from the 0-28th day of testing which was stored at room temperature for each preparation ($p > 0.05$). Good adhesion is indicated by the ease with which the preparation adheres to the applied area. the greater the value of the resulting adhesiveness response means the more time needed for the lipceam preparation to be able to stick to the lips, and vice versa [12]. The results of the stickiness test averaged 4-5 seconds and there was no significant difference from the 0-28th day test which was stored at room temperature for each preparation ($p > 0.05$) [8].

4. CONCLUSIONS

The results of the evaluation of preparations in this study were that conventional lipcream preparations and nanoemulsion lipcream preparations had good stability and there were no significant changes or differences in each evaluation during 28 days of storage at room temperature, but nanoemulsion lipcream preparations were more homogeneous than conventional lipcreams. The color produced by conventional lipcream is more concentrated than nanoemulsion lipcream. In this study, there were limitations in testing nanoparticle samples due to the limited equipment available in oir research laboratory.

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